



NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
WASHINGTON, D.C. 20546

Lewis

(NASA-Case-LEW-11262-1) DEPOSITION OF  
ALLOY FILMS Patent (NASA) 4 p CSCL 11F

N74-1327

REPLY TO  
ATTN OF: GP

00/18 Unclassified  
24224

TO: KSI/Scientific & Technical Information Division  
Attention: Miss Winnie M. Morgan

FROM: GP/Office of Assistant General Counsel for  
Patent Matters

SUBJECT: Announcement of NASA-Owned U.S. Patents in STAR

In accordance with the procedures agreed upon by Code GP and Code KSI, the attached NASA-owned U.S. Patent is being forwarded for abstracting and announcement in NASA STAR.

The following information is provided:

U.S. Patent No. : 3,772,174

Government or Corporate Employee : Government

Supplementary Corporate Source (if applicable) :  

NASA Patent Case No. : LEW-11,262-1

NOTE - If this patent covers an invention made by a corporate employee of a NASA Contractor, the following is applicable:

Yes

No

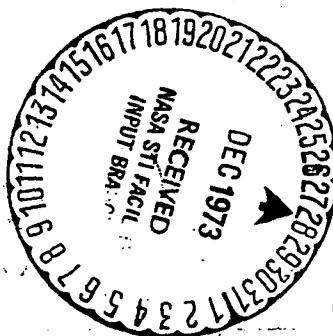
Pursuant to Section 305(a) of the National Aeronautics and Space Act, the name of the Administrator of NASA appears on the first page of the patent; however, the name of the actual inventor (author) appears at the heading of column No. 1 of the Specification, following the words ". . . with respect to an invention of . . ."

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Enclosure

Copy of Patent cited above



**United States Patent** [19]  
**Spalvins**

[11] **3,772,174**  
[45] **Nov. 13, 1973**

[54] **DEPOSITION OF ALLOY FILMS**

[75] **Inventor:** Talivaldis Spalvins, Parma, Ohio

[73] **Assignee:** The United States of America as represented by the Administrator of the National Aeronautics and Space Administration, Washington, D.C.

[22] **Filed:** Apr. 21, 1971

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[52] **U.S. Cl.** ..... 204/192

[51] **Int. Cl.** ..... C23c 15/00

[58] **Field of Search** ..... 204/192, 298

[56] **References Cited**

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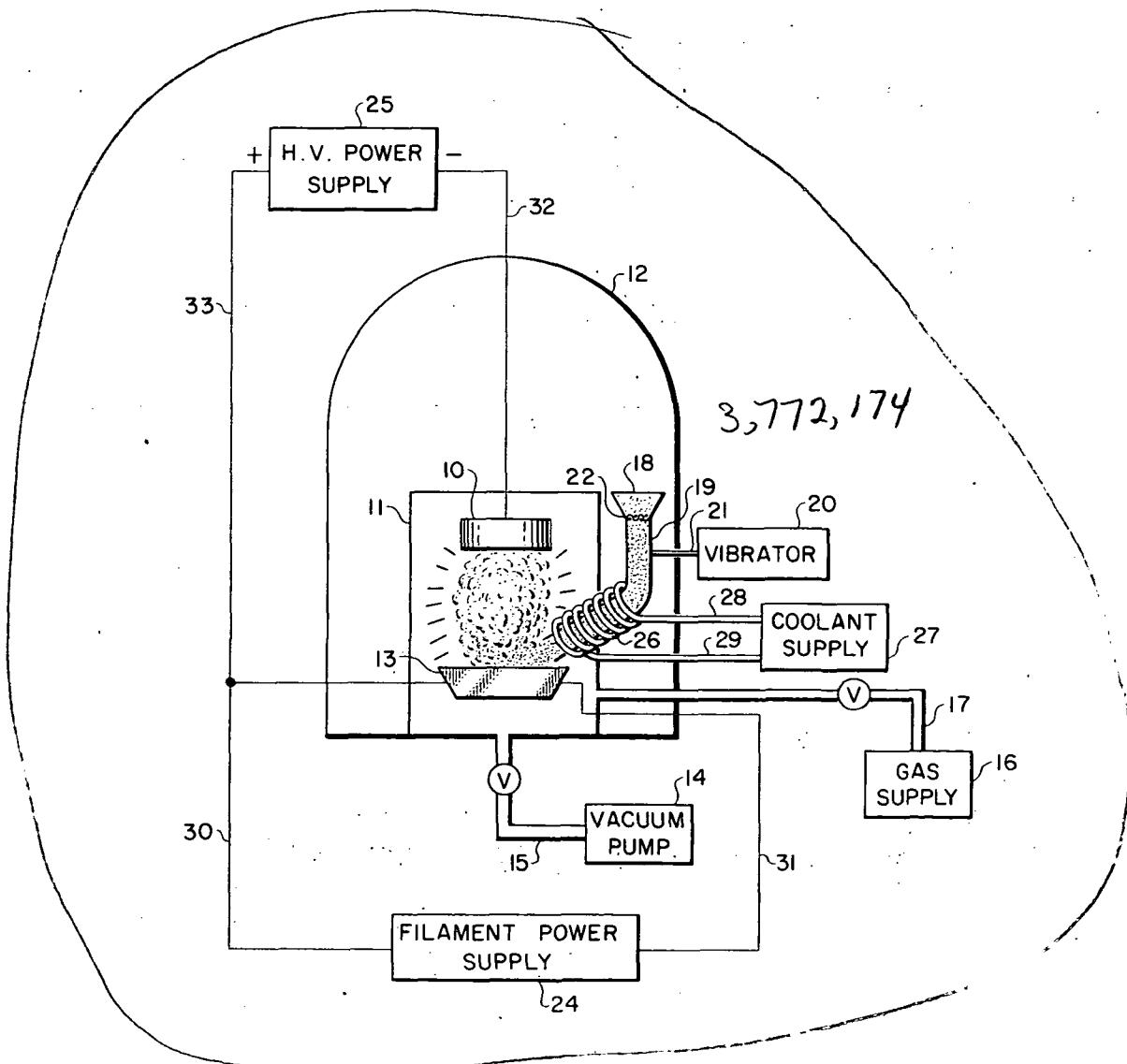
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[57] **ABSTRACT**

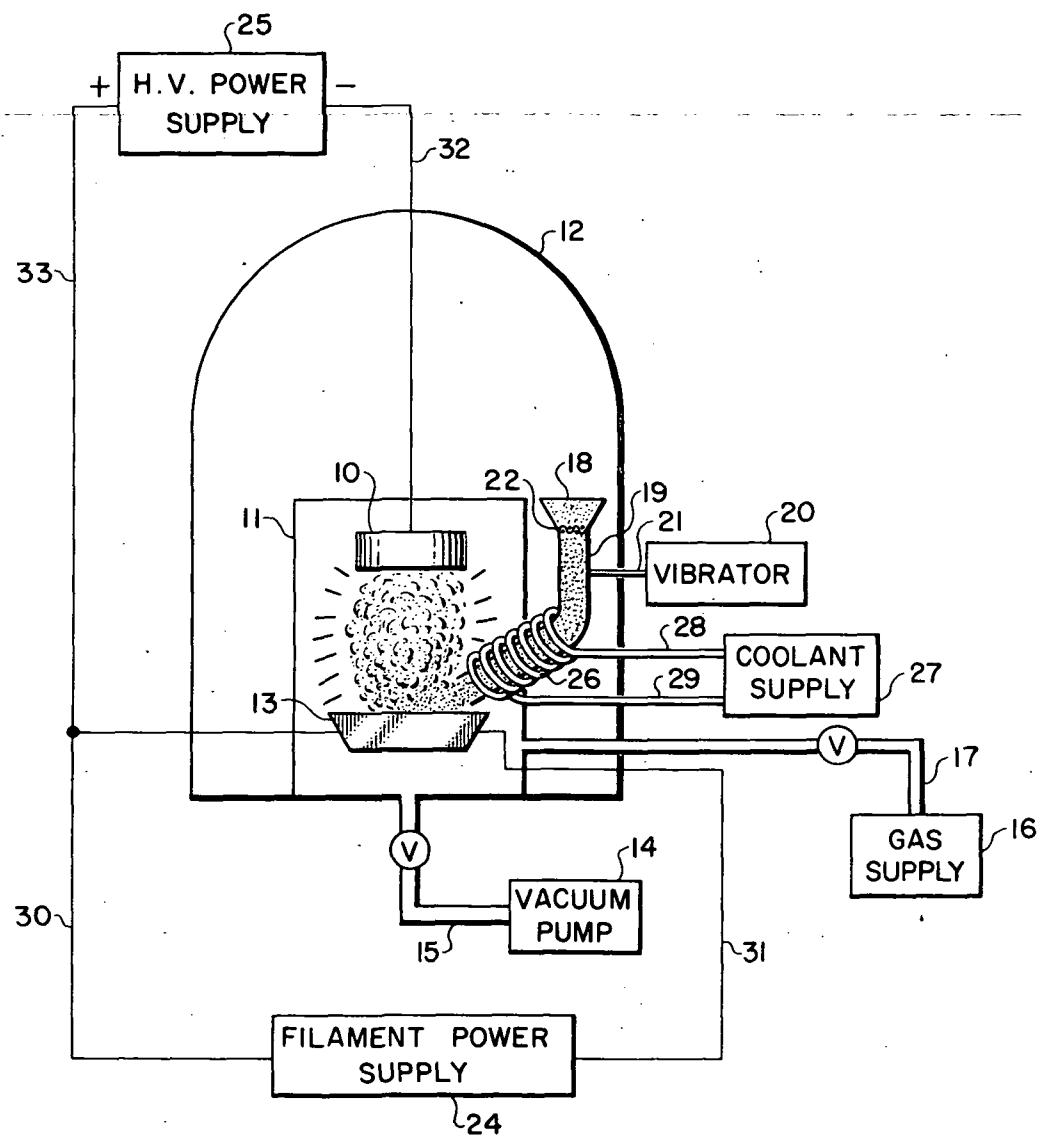
A glow discharge is established by applying a high voltage between an anode and a cathode object disposed in an inert gas atmosphere. An alloy of two or more metals is vaporized and the vapor injected into the glow discharge causing the alloy to be plated onto the cathode object.

7 Claims, 1 Drawing Figure



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3,772,174



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**DEPOSITION OF ALLOY FILMS****ORIGIN OF THE INVENTION**

The invention described herein was made by an employee of the United States Government and may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

**BACKGROUND OF THE INVENTION**

This invention relates to metal coatings and is directed more particularly to a method of depositing metal alloy films on a metal specimen or object.

Metal films have been formed on objects in the past by such techniques as electroplating, vapor deposition, vacuum deposition and ion plating, to name but a few of the most common methods. In general, ion plating techniques can be used to apply an elemental metal coating or film which adheres strongly to the body onto which it is plated. However, alloy coatings have never been successfully plated onto an object by the ion plating technique because the constituent metals of the coating differ significantly percentage-wise from their percentage of weight of the original alloy. Alloys have been successfully deposited by vapor deposition and particularly the flash evaporation technique where an alloy powder vaporizes almost instantaneously as it comes in contact with a heated body and the vapor impinges on the body to be coated. However, the adherence of metal films formed by vapor deposition techniques is low. Furthermore, where the object to be plated has a complex shape the metal film or coating may not be deposited in low spots, cavities, crevices, or the like.

**SUMMARY OF THE INVENTION**

It is an object of the invention to provide a method for applying an alloy coating to a complex-shaped object wherein the alloy coating is formed in a layer of substantially equal thickness in crevices and cavities as well as on high points.

It is another object of the invention to provide a method for forming on an object an alloy coating having an adherence substantially as great as that of a single element formed by ion plating.

Still another object of the invention is to provide a method for producing on an object a metal alloy coating wherein the percentage weight of each element in the coating is substantially the same as the percentage weight of each element in the source alloy.

**DESCRIPTION OF THE DRAWING**

The single FIGURE is a schematic drawing of the apparatus utilized in performing the inventive method.

**DESCRIPTION OF THE PREFERRED EMBODIMENTS**

In accordance with the method of the invention, a metal specimen or object 10 which is to be coated with an alloy is placed in a quartz cylinder 11, the quartz cylinder 11 being inside an airtight bell jar 12. A container or boat 13 of tungsten, molybdenum or tantalum is disposed below the object 10 in the quartz cylinder 11. The boat may be replaced by an electrical resistance filament if desired. The interior of the quartz cylinder 11 and the bell jar 12 are evacuated by means of a vac-

uum pump 14 which communicates with the interior of the quartz cylinder 11 by means of a conduit 15. The interior of the quartz cylinder 11 is then purged with an inert gas such as argon supplied from a gas supply 16 through a conduit 17. The inert gas, under pressure, is retained in the quartz cylinder 11 and the bell jar which communicates with the quartz cylinder.

A powder of the alloy to be deposited on the object 10 is disposed in the upper end 18 by a delivery tube 19 which extends into the quartz cylinder 11 terminating above the boat 13. Upon activation of a vibrator 20, which is mechanically connected to the delivery tube 19 by a rod 21, the powdered alloy passes through a mesh or screen 22 and is deposited on the upper surface of the boat 13.

To the end that the alloy powder will vaporize substantially instantaneously upon contact with the boat 13, the electrical resistance of the boat is utilized by connecting the boat to a suitable floating output power supply 24 via leads 30 and 31. The boat temperature must be such that the elemental metal of the alloy having the highest vaporization temperature will be vaporized substantially instantaneously.

In order that the object 10 and the boat 13 may serve as a cathode and an anode, respectively, to establish a glow discharge in the inert gas atmosphere contained in the quartz cylinder 11, the object 10 is connected to the negative side of a high voltage power supply 25 via a lead 32 while the boat 13 is connected to the positive side of the high voltage power supply via a lead 33.

In accordance with the method of the instant invention a glow discharge is maintained between the object 10 and the boat 13 for a length of time sufficient to clean the surface of the object by the etching effect

which results from the glow discharge. The boat is heated to a prescribed temperature during the time that the object is being etched. When the object 10 is sufficiently clean, the vibrator is activated causing the alloy powder to pass through the delivery tube 19 and be delivered to the boat 13. When the alloy powder contacts the hot surface of the boat 13, the powder vaporizes substantially instantaneously. This alloy vapor is injected into the glow discharge region present between the object 10 and the boat 13. This causes the alloy to plate onto the object 10 forming a diffusion bond therewith. If the object 10 includes crevices and cavities, the alloy will advantageously plate these areas. If desired, a cooling coil 26 may be disposed around the delivery tube 19 to maintain the alloy powder at a prescribed temperature before it impinges on the boat 13. The coil 26 may be connected to a suitable coolant supply 27 by means of tubes 28 and 29.

Examples of some typical parameters to be used with the method of the instant invention will now be given:

**EXAMPLE I**

Alloy powder and weight percent:—Pb-Sn(50-50)

Object to cathode distance:—14 cm

Argon discharge pressure:—20  $\mu$

Cathode to anode voltage:—5 kv

Particle size:—125  $\mu$

Screen (22):—120 mesh

Boat temperature:—2100–2350°F

**EXAMPLE II**

Parameters are the same as for Example I except the percent weight ratio of the Pb-Sn alloy is 34–65.

## EXAMPLE III

Alloy powder and weight percent: Cu-Au (85-15)  
 The other parameters are the same as for Examples I and II except for the boat temperature which is in a range of from about 3050°F to about 3150°F with the preferred temperature being about 3100°F.

Analysis of the alloy coatings by spectrographic analysis, X-ray fluorescence and wet chemical analysis indicates that the percent weight of each element of the coating is substantially the same as that for each element of the initial alloy powder as shown in Table I below:

TABLE I

Alloy	Original Composition weight %	Composition of film weight %
Pb-Sn	48.24-48.10	49.61-50.39
Pb-Sn	33.87-65.42	36.78-64.20
Cu-Au	84.32-15.17	85.1-14.8

It will be understood that each parameter must be adjusted in accordance with the other parameters being involved. For example, the vibrator must be adjusted to provide a delivery rate for the alloy powder such that the powder does not build up or accumulate on the surface of the boat. This of course depends on the temperature of the boat. The length of time of the coating operation is selected to produce the desired coating thickness. If the anode (boat) to cathode (object) distance is increased, it will be understood that the high voltage must also be increased.

In view of the foregoing, it will be seen that there is provided a method wherein an alloy vapor is injected into a glow discharge region and deposits an alloy coating on an object or specimen. The coating will be applied to inside crevices and cavities as well as on raised portions of the specimen and will adhere strongly to the surface. Furthermore, the composition of the alloy coating will be substantially the same as the composition by percentage weight of the alloy metal used to provide the coating.

It will be understood that the invention may be changed or modified by those skilled in the art without

departing from the spirit and scope of the invention as set forth in the claims appended hereto.

What is claimed is:

1. A method of plating a complex shaped metal object with an alloy comprising the steps of

disposing said metal object with a second metal body including flash evaporating means in a chamber containing an inert gas;

applying a high voltage between said object and said second body to establish a glow discharge, said first metal object being negative with respect to said second metal object;

maintaining said glow discharge to etch the surface of said metal object;

supplying an alloy powder having a particle size ranging from about 100  $\mu$  to 300  $\mu$  to said flash evaporating means at a predetermined rate;

flash vaporizing said alloy; and

directing the vapor into said glow discharge whereby said alloy plates said metal object.

2. The method of claim 1 wherein said alloy is vaporized by heating said second metal body and by depositing said powder of said alloy on said second metal body at said predetermined rate.

3. The method of claim 2 wherein said alloy powder is Pb-Sn and said second metal body is heated to a temperature in the range of from about 2100°F to about 2350°F.

4. The method of claim 3 wherein said inert gas is argon at a pressure of about 20  $\mu$ .

5. The method of claim 2 wherein said alloy powder is Cu-Au and said second metal body is heated to a temperature in the range of from about 3050°F to about 3150°F.

6. The method of claim 5 wherein said inert gas is argon at a pressure of 20  $\mu$ .

7. The method of claim 1 wherein said high voltage is in the range of from about 3 kv to 5 kv; said inert gas is argon at a pressure of about 20  $\mu$ ; and the distance between said first and second metal bodies being about 14 c.m.

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